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An experimental analysis on property and structure variations of agricultural wastes undergoing torrefaction

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HIGHLIGHTS

- ▶ Coffee residue, sawdust and rice husk undergoing torrefaction are investigated.
- ▶ A high-volatile bituminous coal and a low-volatile one are also regarded for comparison.
- ▶ Coffee residue is the most active biomass to torrefaction.
- ▶ Torrefied wastes approach the high-volatile coal as increasing temperature and duration.
- ▶ Torrefaction is conducive to the applications of biomass in industrial furnaces.

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ABSTRACT

Three agricultural wastes, consisting of coffee residue, sawdust and rice husk, undergoing torrefaction are investigated to evaluate the potential of biomass as solid fuel. Two different torrefaction temperatures (240 and 270 °C) and durations (0.5 and 1 h) are considered in the study, and the properties and structures of the raw and torrefied wastes are extensively investigated by means of proximate, elemental, fiber, calorific, thermogravimetric, SEM and FTIR analyses. A high-volatile bituminous coal and a low-volatile one are also regarded for comparison. By virtue of more hemicellulose contained in the coffee residue, it is the most active biomass to torrefaction and its higher heating value (HHV) is improved up to 38%. The empirical atomic formula of the raw wastes is expressed by $CH_{1.54-1.76}O_{0.65-0.89}$ and it changes to $CH_{1.02-1.57}O_{0.26-0.64}$ after undergoing torrefaction. The torrefied biomasses approach high-volatile coal when the torrefaction temperature and duration increase. From fuel point of view, the improved properties and changed molecular structure are conducive to the applications of biomass in industrial furnaces such as boilers and blast furnaces.

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1. Introduction

According to the statistics of International Energy Agency (IEA), the development of renewable energy in the world has grown at an average annual rate of 1.7% [1]. Renewable energy sources include solar, wind, biomass, geothermal, hydropower and ocean energies. In these renewable energy sources, biomass is the largest one which approximately accounts for 10% of the world's annual energy consumption in comparison to 21%, 27% and 33% from natural gas, coal and petroleum oil, respectively [2]. In currently developing bioenergy systems, biomass can be consumed directly from the solid state; it can also be converted into liquid biofuels and gas fuels such as biogas or synthesis gas (syngas). When one is concerned with solid biomass, it is always desirable to utilize agricultural wastes, which are abundant worldwide, as alternatives to fossil fuels [3]. For example, it has been reported that Taiwan produced around 5 million metric tons of agricultural wastes in 2009 [4].

Biomass has been considered as a carbon neutral fuel in that the net carbon emissions from burning biomass are zero. However, raw biomass is generally characterized by its higher moisture content and volatile matter as well as lower heating value and energy density [5] compared to fossil fuels. Besides, it is difficult to grind biomass into small particles stemming from its lignocellulosic nature. By virtue of higher volatile matter in raw biomass, this results in less char to be produced and burned in combustors. For these reasons, the potential of raw biomass serves as an alternative fuel of coal is restricted. Nevertheless, the aforementioned drawbacks can be resolved through the pretreatment methods. Among the pretreatment methods, torrefaction is a promising technique to upgrade solid biomass [6–10]. Torrefaction is a thermal pretreatment process where

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raw biomass is heated in an inert atmosphere at the temperature range of 200–300 °C [11–13], and nitrogen is the most commonly used purge gas to provide a non-oxiding environment.

It is well known that the principal and basic constituents in biomass are hemicellulose, cellulose and lignin [14,15]; small amount of extractives are also contained in biomass. Hemicellulose is a branched polymer, whereas cellulose is a straight chain polymer. Both hemicellulose and cellulose are densely packed by layers of lignin. On account of inherent difference in the structures of hemicellulose, cellulose and lignin, their decomposition temperatures are in the ranges of 200–315, 315–400 and 160–900 °C, respectively [16]. Accordingly, it has been reported that torrefaction has a significant impact upon hemicellulose, whereas cellulose and lignin are affected by the pretreatment to a certain extent, depending mainly on torrefaction temperature and duration [7]. Some studies have outlined the impact of torrefaction temperature on the properties of torrefied biomass. For example, in the study of Yan et al. [17], they pointed out that an increase in torrefaction temperature resulted in a decrease in the mass yield of loblolly pine, but led to an increase of carbon content in the treated solid biomass. Chen et al. [18] investigated the torrefaction of a woody biomass at the conditions of light (220 °C), mild (250 °C) and severe (280 °C) torrefactions. They highlighted that the severe torrefaction was able to increase the calorific value of the wood up to 40%. But over 50% of mass was lost from the pretreatment. Rousset et al. [19] also studied bamboo torrefied at the three different temperatures. They found that the energy yield was larger than the mass yield and increasing temperature intensified this difference.

Reviewing past studies indicates that when torrefaction is employed to pretreat biomass, some of the oxygen and hydrogen in raw biomass are consumed from the thermal degradation. Meanwhile, biomass experiences partial carbonization. The O/C and H/C ratios in torrefied biomass are reduced, thereby increasing its heating value [17,20,21]. It has also been reported that the ignitability, reactivity and grindability of torrefied biomass are better compared to their parent one [20,22,23]. The advantages of torrefaction have been addressed in a number of published papers. However, some information from the torrefaction of solid agricultural wastes remains insufficient, especially in their molecular structures. To provide a deep insight into the technique of torrefaction, this study is intended to investigate the impact of torrefaction temperature and duration upon the properties of three agricultural wastes. Particular emphasis is placed on the variations of ultimate analysis results and internal structure of lignocelluloses due to the reactions of hemicellulose, cellulose and lignin.

2. Experimental

2.1. Reaction system

The schematic of the reaction system is displayed in Fig. 1. The experimental system was made up of a nitrogen cylinder, a rotameter, a reactor and a product gas treatment unit. The steel cylinder was used to supply nitrogen for providing a non-oxidizing environment. The volumetric flow rate of nitrogen was controlled by the rotameter. The reactor comprised a quartz tube, a tube furnace and a temperature controller. Tested samples were placed in the tube which was situated in the furnace. A temperature probe (thermocouple) was mounted in the furnace beside the quartz tube to detect the temperature. The power of the furnace was controlled by the temperature controller. A conical flask was employed as the product gas treatment unit to remove tars and clean the exhaust gas stemming from torrefaction.

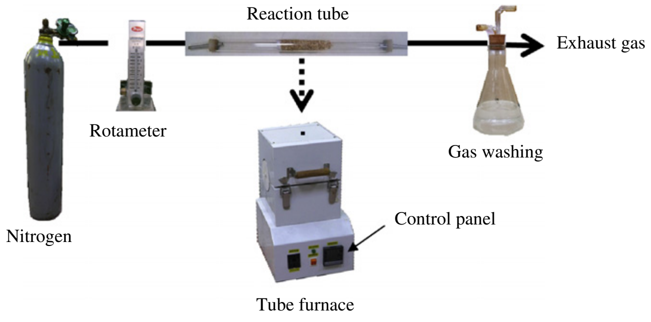


Fig. 1. A schematic of experimental system.

2.2. Experimental procedure and analyses

Three different agricultural wastes, including coffee residue, sawdust and rice husk, were tested. The coffee residue was obtained from coffee beans; the sawdust was from the sawing of woods and the rice husk was collected from the treatment of rice in Taiwan. The wastes were preliminarily ground by a shredder and sieved by vibrating screens. On account of intrinsic difference in the wastes, the particle sizes of the coffee residue and sawdust were controlled between 100 and 200 mesh (i.e. 75–150 μm). On the other hand, the particle sizes of the rice husk were located between 40 and 100 mesh (i.e. 150–385 μm). The sieved biomasses were dried in an oven at 105 °C for 24 h to prepare the experimental samples. Subsequently, the samples were placed

Table 1
Proximate analysis and fiber analysis of three solid wastes (wt.%).

Raw material	Coffee residue	Sawdust	Rice husk
Proximate analysis			
Moisture	14.50	12.90	11.51
Volatile matter (VM)	68.80	61.36	61.13
Fixed carbon (FC)	14.94	13.96	16.37
Ash	1.76	11.78	10.99
Fiber analysis			
Hemicellulose	40.10	11.18	21.34
Cellulose	27.38	36.08	36.06
Lignin	6.00	28.66	21.16
Other	26.52	24.08	21.44

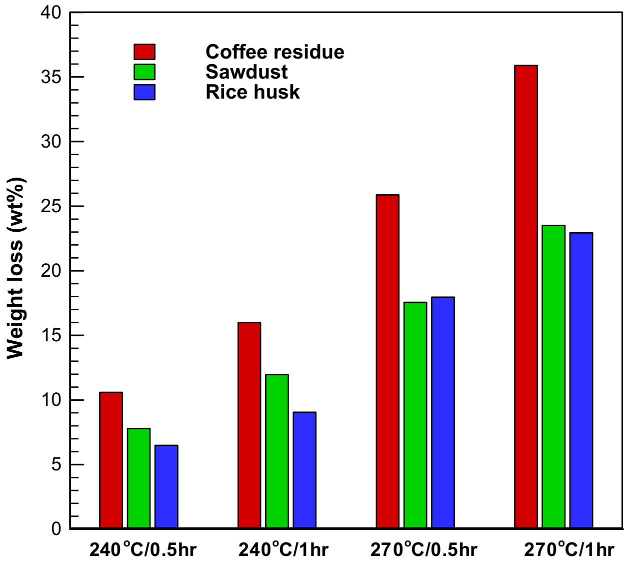


Fig. 2. Weight loss distributions of tested solid wastes at various torrefaction temperatures and durations.

Table 2
Elemental analyses, ash content, O/C and H/C atomic ratios as well as their formulas of raw and torrefied wastes.

Material	Temp (°C)	Time (h)	Elemental analysis (wt.%)					Ash	O/C	H/C	$C_xH_yO_z$
			C	H	N	O	S				
Coffee residue											
	Raw material		47.45	6.91	2.55	41.09	0.24	1.76	0.65	1.75	$CH_{1.75}O_{0.65}$
	240	0.5	50.83	6.66	2.74	37.74	0.06	1.97	0.56	1.57	$CH_{1.57}O_{0.56}$
	240	1	52.10	6.36	2.57	36.43	0.09	2.45	0.52	1.46	$CH_{1.46}O_{0.52}$
	270	0.5	55.93	5.60	2.67	32.52	0.07	3.21	0.44	1.20	$CH_{1.20}O_{0.44}$
	270	1	65.10	5.52	2.94	22.50	0.08	3.86	0.26	1.02	$CH_{1.02}O_{0.26}$
Sawdust											
	Raw material		42.56	5.46	2.85	37.23	0.12	11.78	0.66	1.54	$CH_{1.54}O_{0.66}$
	240	0.5	42.49	5.34	2.80	33.08	0.10	16.19	0.58	1.51	$CH_{1.51}O_{0.58}$
	240	1	43.46	5.25	2.93	31.57	0.09	16.70	0.54	1.45	$CH_{1.45}O_{0.54}$
	270	0.5	43.48	4.78	3.14	30.12	0.08	18.40	0.52	1.32	$CH_{1.32}O_{0.52}$
	270	1	48.67	4.79	3.18	22.99	0.06	20.31	0.35	1.18	$CH_{1.18}O_{0.35}$
Rice husk											
	Raw material		38.15	5.60	0.13	45.07	0.06	10.99	0.89	1.76	$CH_{1.76}O_{0.89}$
	240	0.5	44.19	5.35	0.10	37.81	0.03	12.52	0.64	1.45	$CH_{1.45}O_{0.64}$
	240	1	44.28	5.32	0.09	37.46	0.00	12.85	0.63	1.44	$CH_{1.44}O_{0.63}$
	270	0.5	46.59	5.15	0.09	33.76	0.02	14.39	0.54	1.33	$CH_{1.33}O_{0.54}$
	270	1	48.15	4.75	0.11	31.26	0.00	15.73	0.49	1.18	$CH_{1.18}O_{0.49}$
Low-volatile coal			84.24	3.82	1.64	1.88	0.53	7.89	0.02	0.54	$CH_{0.54}O_{0.02}$
High-volatile coal			76.15	5.15	1.61	8.10	0.49	8.50	0.08	0.81	$CH_{0.81}O_{0.08}$

in sealed plastic bags and stored in a desiccator at room temperature until experiments were carried out. In each experiment, 10 g of sample was packed at the center of the quartz tube. The tube furnace was first heated to the desired torrefaction temperature. Then, the tube was placed in the furnace. While tested sample was torrefied, nitrogen at the flow rate of 100 mL min^{-1} (25 °C) was continuously blown into the reaction tube to keep the thermal degradation of biomass in an inert environment and to remove volatiles liberated from the tested materials. Soon after the gas mixture left the tube, it was cooled and washed by water which was filled in the conical flask so as to clean the exhaust gas.

Prior to performing experiments, the reaction system was leak tested using nitrogen to ensure the measurement quality. The experiment under any given condition was usually carried out more than twice. The results were fairly uniform between each run and the relative error was controlled below 5%. All data in the study were averaged from the measurements. Before and after the wastes undergoing torrefaction, their properties were extensively analyzed. The measurements of the properties of the raw and torrefied samples include proximate, elemental or ultimate (Elementar Vario EL III), fiber, calorific (IKA C2000 Basic), thermogravimetric (PerkinElmer Diamond TG/DTA), scanning electronic microscopic (FEI Quanta 400F) and Fourier transform infrared (PerkinElmer/Spectrum 100) analyses.

The proximate analysis was performed in accordance with the standard procedure of American Society for Testing and Materials (ASTM). Fiber analysis (hemicellulose, cellulose and lignin) were analyzed following the method adopted in a previous study [13], that is, they were determined from the measurements of neutral detergent fiber, acid detergent fiber and ash. The non-isothermal TGA was executed with the heating rate of 20 °C min^{-1} and the N_2 flow rate of 100 mL min^{-1} where the temperature ranged from 25 to 800 °C. When the temperature reached 105 °C, it was held for 10 min to ensure moisture removal. In Fourier transform infrared analysis, three standards of hemicellulose, cellulose and lignin, were employed for comparison; they were purchased from SIGMA (X4252), Alfa Aesar (A17730) and Tokyo Chemical Industrial Co. (L0045), respectively.

3. Results and discussion

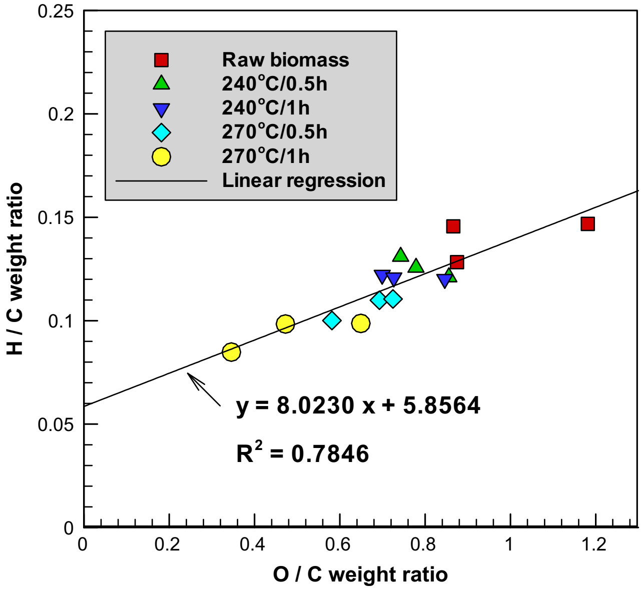
In the experiments, two torrefaction temperatures of 240 and 270 °C and two durations of 0.5 and 1 h are regarded. The basic properties of the three wastes such as proximate and fiber analyses are listed in Table 1. It is worthy of note that the properties of the coffee residue are different from the other two. Specifically, the coffee residue possesses the lowest ash and lignin contents (i.e. 1.76 and 6.00 wt.%), whereas it has the highest hemicellulose content (i.e. 40.10 wt.%). Meanwhile, the sawdust included bark so its ash content was higher (i.e. 11.78 wt.%).

3.1. Variations of weight and elements of solid wastes

The influences of torrefaction temperature and duration on the weight loss of the three wastes are first demonstrated in Fig. 2. Overall, within the investigated ranges of the operating conditions, increasing temperature or duration has a pronounced trend to intensify weight loss. In particular, the impact of torrefaction at 270 °C for 0.5 h on the three wastes is more than that at 240 °C for 1 h. The coffee residue is the most sensitive species to torrefaction among the three wastes, regardless of what the temperature and duration are. Specifically, when the torrefaction temperature is 270 °C and the duration is 1 h, around 23 wt.% of sawdust or rice husk is lost, whereas it is 36 wt.% for the coffee residue. This is attributed to higher volatile matter and hemicellulose contained in the coffee residue (Table 1) where the thermal degradation of hemicellulose in a torrefaction environment is more drastic than those of cellulose and lignin [7,8]. Besides, lignin contained in the coffee residue is less than that in the sawdust or rice husk where lignin has a higher resistance to thermal degradation.

The weight percentages of elements C, H, N, O and S as well as ash content of the raw and torrefied solid wastes are listed in Table 2. When the pretreatment is carried out, the weight percentage of C in biomass increases with increasing torrefaction temperature and duration. On the contrary, the weight percentages of H and O have a trend to decrease. It is worth noting that the weight percentage of S also decays when the temperature or duration increases. Based on the weight percentage shown in Table 2, the

(a) Weight ratio



(b) Atomic ratio

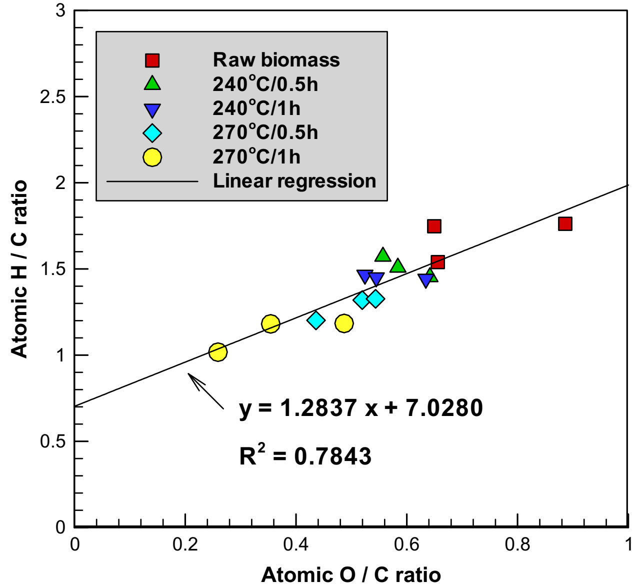


Fig. 3. Distributions of (a) H/C versus O/C weight ratio and (b) atomic H/C versus O/C ratio.

profiles of H/C ratio versus O/C ratio in terms of weight and atom are plotted in Fig. 3a and b, respectively. It is clear that the profiles are characterized by a linear relationship. The slope of the regression line (=1.2837) shown in Fig. 3b reflects that the influence of torrefaction on hydrogen is more than on oxygen. The atomic O/C ratios of the three raw wastes are approximately located between 0.65 and 0.89 and their atomic H/C ratios range from 1.54 to 1.76. When the wastes undergo torrefaction, the atomic O/C and H/C ratios are in the ranges of 0.26–0.64 and 1.02–1.57, respectively.

Furthermore, the relative mole fractions of C, H and O in the raw wastes, torrefied biomasses and coals are shown in Fig. 4 using a triangular diagram in which a low-volatile bituminous coal and a high-volatile one [24] are included for comparison. Based on the mole fractions, the empirical atomic formulas of the raw and torrefied wastes are expressed in Table 2 as well.

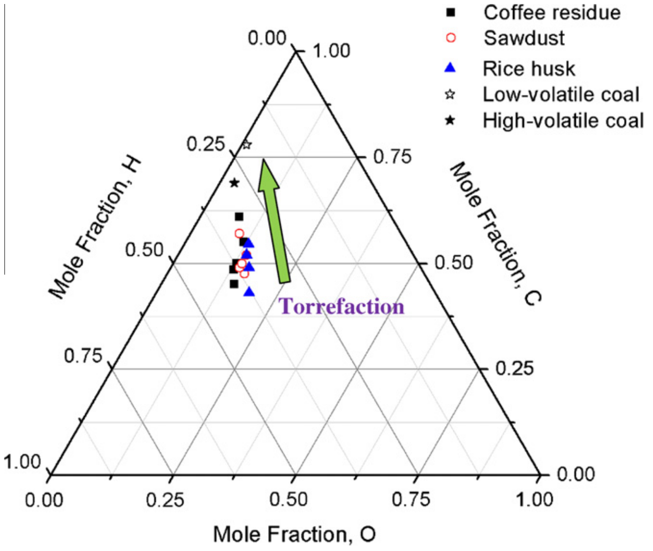


Fig. 4. Profiles of relative mole fraction of elements C, H and O of raw and torrefied solid wastes under various torrefaction temperatures and durations.

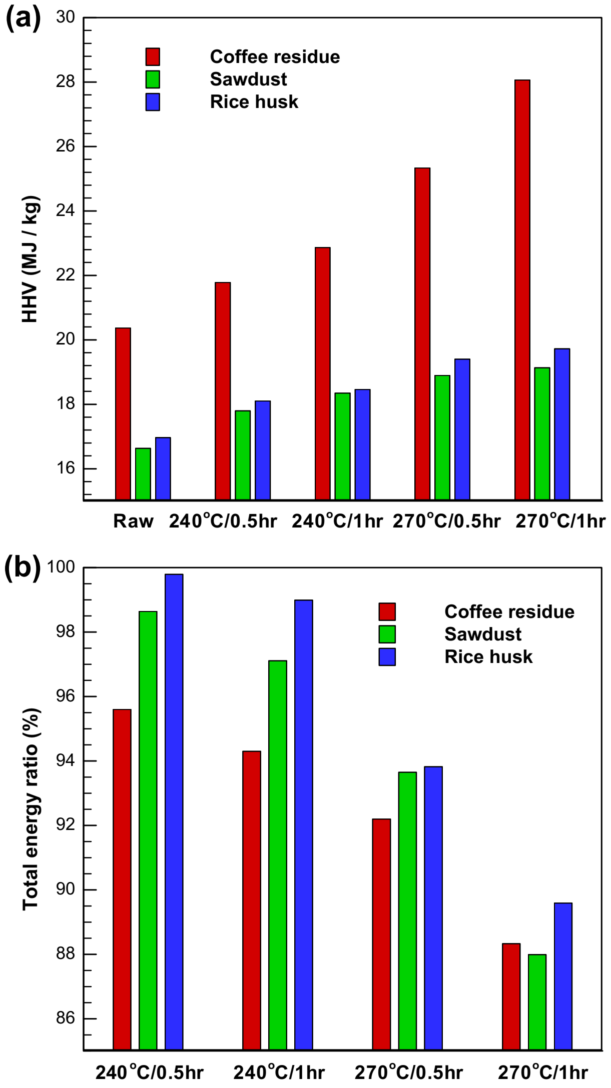
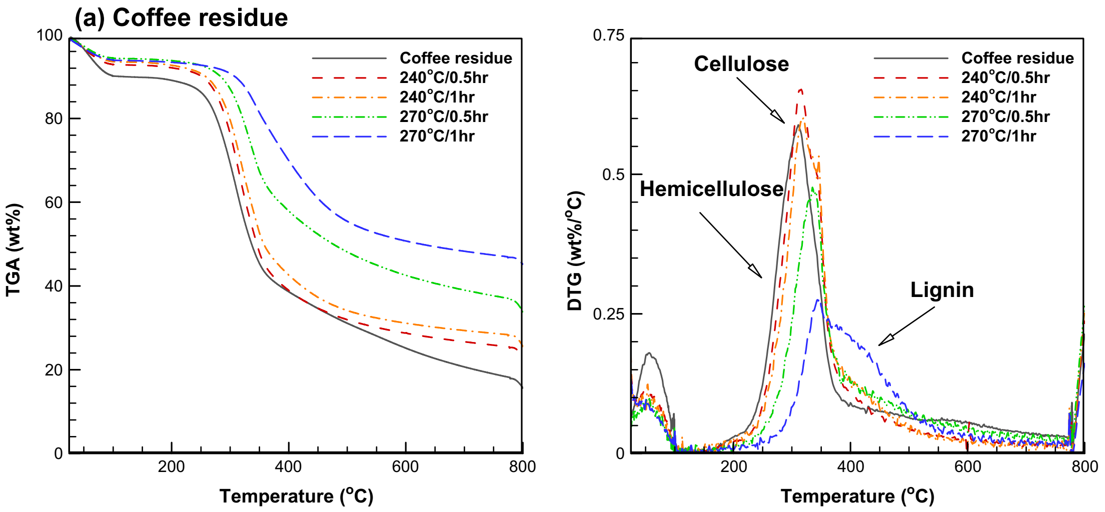
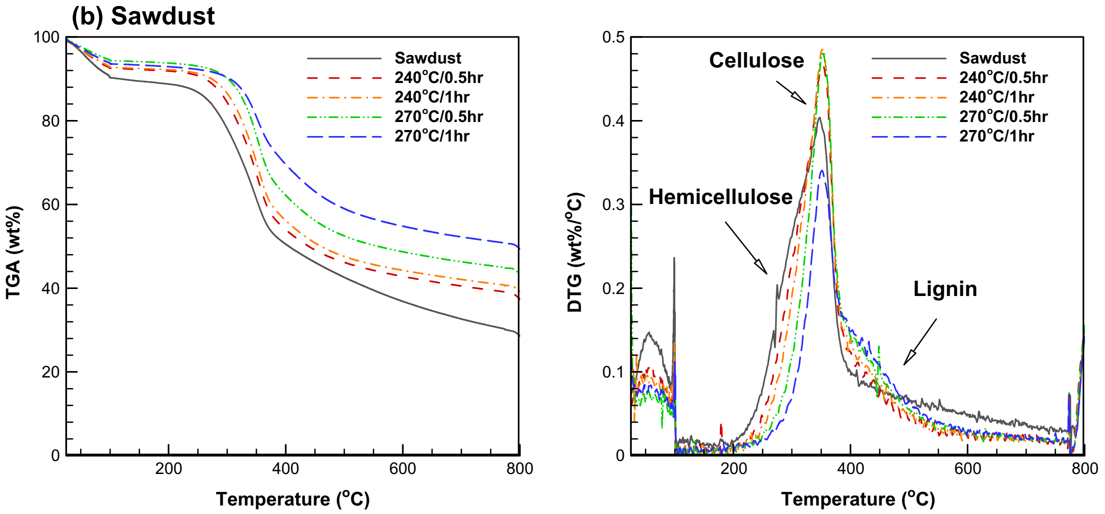


Fig. 5. Distributions of (a) higher heating value (HHV) and (b) energy yield of tested solid wastes at various torrefaction temperatures and durations.


(a) Coffee residue


(b) Sawdust

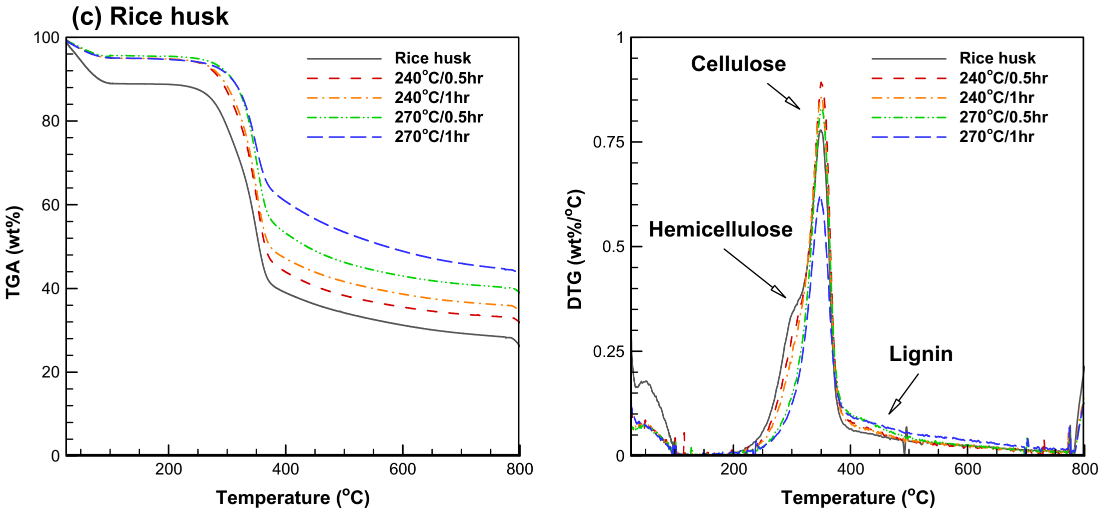

(c) Rice husk

Fig. 6. Distributions of TGA and DTG of raw and torrefied (a) coffee residue, (b) sawdust and (c) rice husk.

From these formulas, it can be seen that atomic carbon contained in the wastes is higher than atomic oxygen but it is less than atomic hydrogen. When the two coals are examined, the empirical atomic formulas of the low-volatile coal and the high-volatile one can be expressed as $CH_{0.54}O_{0.02}$ and $CH_{0.81}O_{0.08}$, respectively. This suggests that atomic carbon is always higher than the atomic hydrogen; alternatively, atomic oxygen is fairly low in the coals. For the wastes, once the torrefaction temperature and duration are higher, the torrefied biomasses are more approaching the high-volatile coal, as shown in Fig. 4. This reflects that biomass undergoing severe torrefaction has the potential as the fuel to replace high-volatile coals.

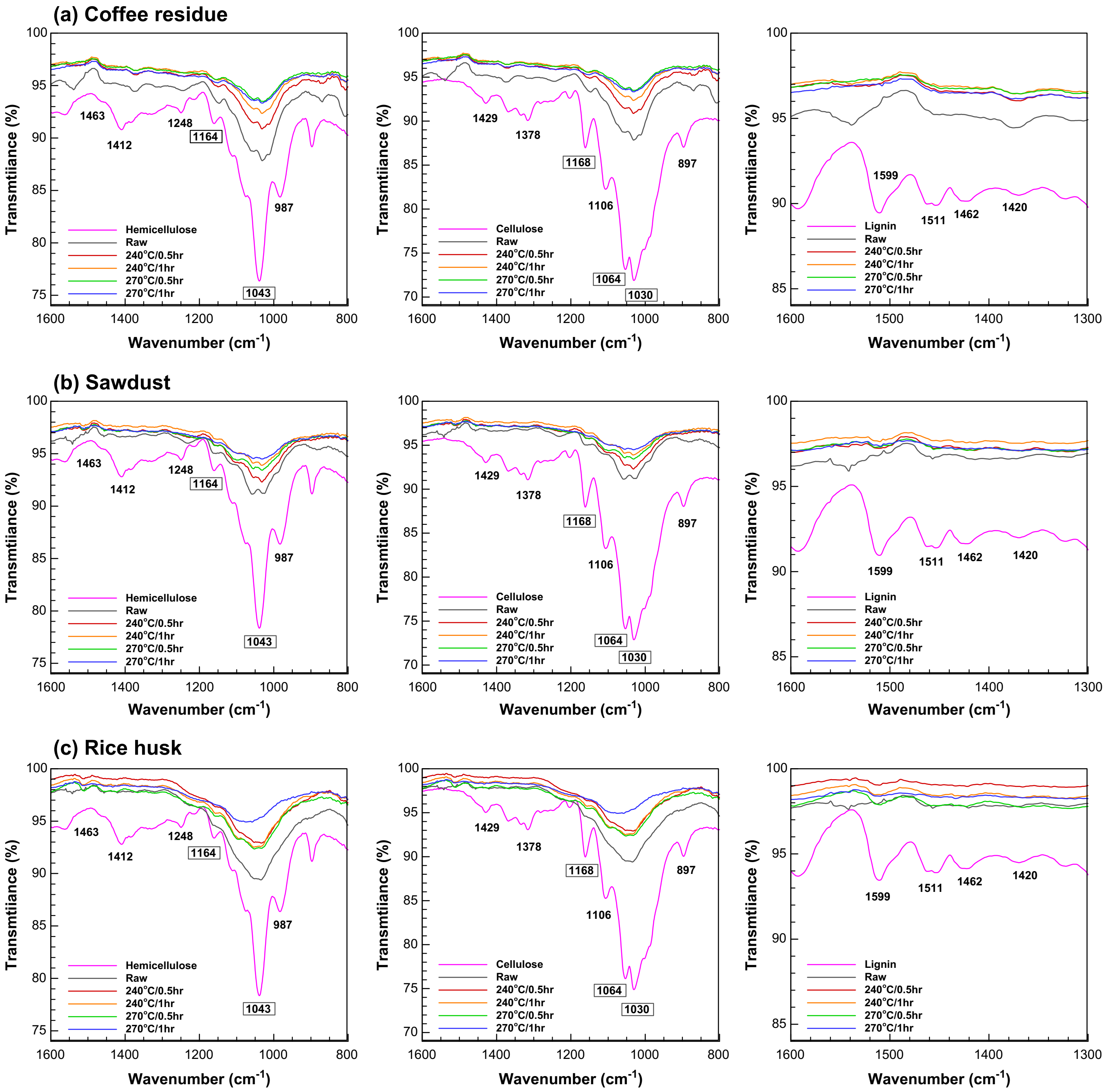


Fig. 7. FTIR spectra of raw and torrefied (a) coffee residue, (b) sawdust and (c) rice husk.

3.2. Heating value and energy yield

Fig. 5a and b displays the distributions of the higher heating value (HHV) and energy yield of the three solid wastes, respectively. The energy yield is defined by:

$$\text{Energy yield }(\%) = \frac{\text{Total energy of torrefied biomass}}{\text{Total energy of raw biomass}} \times 100 = \frac{\text{HHV}_{\text{torrefied biomass}} \times \text{weight of torrefied biomass}}{\text{HHV}_{\text{raw biomass}} \times \text{weight of raw biomass}} \times 100 \qquad (1)$$

It can be seen that the HHV of the raw wastes is in the range of 16–21 MJ kg^{-1}. Once they undergo torrefaction, their HHV is enlarged to 18–28 MJ kg^{-1}, especially for the conditions of 270 °C and 1 h. A comparison among the three wastes, Fig. 5a indicates that the coffee residue is relatively sensitive to the torrefaction time and duration in that its HHV grows substantially with increasing temperature and duration. In Fig. 2, it has been observed that the weight loss of the coffee residue is more drastic than the other two, as a consequence of more hemicellulose contained. It follows that the higher the hemicellulose in biomass, the more pronounced the HHV of biomass enhanced from torrefaction. The weight loss will lessen energy yield, whereas the enhancement of HHV facilitates energy yield. Seeing that the impact of the former on energy yield is over the latter, the energy yield decreases with increasing temperature and duration. It is clear that around 96–99% of energy is retained in the wastes

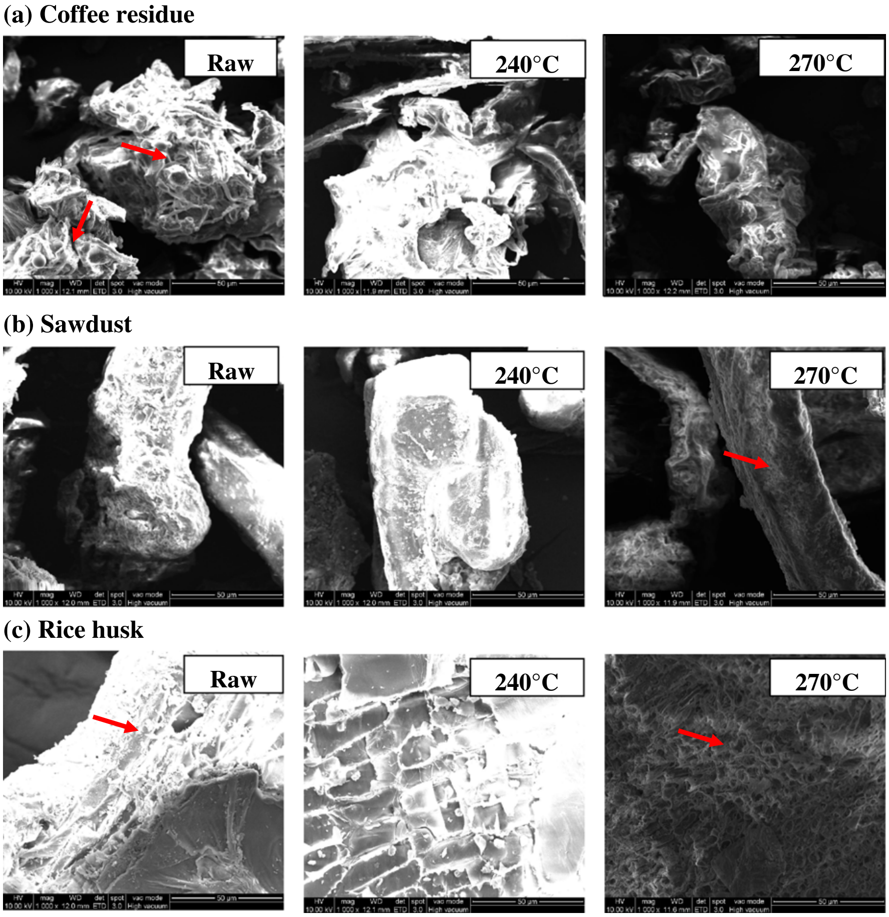


Fig. 8. SEM images of raw and torrefied (a) coffee residue, (b) sawdust and (c) rice husk with duration of 1 h.

when they are treated at 240 °C for 0.5 h. In contrast, the energy yield decays to 88–90% when the torrefaction temperature and duration are 270 °C and 1 h, respectively.

3.3. Thermogravimetric analysis (TGA)

To proceed farther into an analysis of the impact of torrefaction are upon the wastes, the results of thermogravimetric analyses (TGA) and derivative thermogravimetric (DTG) analyses are plotted in Fig. 6. When the temperature in the thermogravimetry is between 25 and 105 °C, it is noted that the weight drop of the raw wastes is more pronounced than that of the torrefied counterparts, verifying that the torrefied wastes are characterized by the hydrophobic behavior. The thermal decompositions of the raw coffee residue, sawdust and rice husk mainly occur within the temperature ranges of 240–320, 200–360 and 240–360 °C, respectively, and the thermal degradations of hemicellulose, cellulose and lignin can be clearly identified. For the coffee residue, the impact of torrefaction with 240 °C on the lignocellulosic structure is relatively slight in that the shrinkage of the peak is not obvious (Fig. 6a). With the torrefaction temperature of 270 °C, the peak withers to a great extent, especially for the duration of 1 h. This implies that the torrefaction with the aforementioned conditions has a marked impact upon the lignocellulosic structure, stemming from the thermal degradations of hemicellulose and cellulose. In examining the DTG curves of the sawdust and rice husk (Fig. 6b and c), the thermal degradations of hemicellulose and cellulose can be identified [25]. Once the two wastes experience torrefaction, the peaks of hemicellulose disappear. Unlike the coffee residue, the influence of torrefaction at 270 °C with 1 h duration on the lignocellulosic structures of the sawdust and rice husk is not as significant as on the coffee residue, revealing that the resistance of the sawdust and rice husk to torrefaction at a higher temperature with longer duration is higher than that of the coffee residue.

3.4. Fourier transform infrared spectra

The Fourier transform infrared spectrometry (FTIR) is also used to realize the impact of torrefaction upon lignocellulosic structure. As shown in Fig. 7, the peaks at 1463, 1412, 1248, 1164, 1043 and 987 cm^{-1} are the typical absorption bands of hemicellulose [26]. The typical absorption bands of cellulose occur at 1429, 1378, 1168, 1106, 1064, 1030 and 897 cm^{-1} [27], whereas the typical absorption bands of lignin are at 1599, 1511, 1462, and 1420 cm^{-1} [3,28]. Obviously, the FTIR spectra at the bands around 1043 and 1164 cm^{-1} indicate that increasing torrefaction temperature progressively consume hemicellulose in the wastes. Most of the hemicellulose contained in the three wastes is consumed when the torrefaction temperature of 270 °C is carried out. Alternatively, the absorption bands at 1030, 1064 and 1168 cm^{-1} suggest that cellulose can be identified in the raw wastes; but the peaks disappear when they are torrefied, especially for the conditions of 270 °C and 1 h duration. With regard to lignin, it is difficult to be identified in the wastes, regardless of the raw or torrefied wastes are examined.

3.5. Scanning electronic microscope

Eventually, the scanning electron microscope (SEM) is used to observe the morphology of the three solid wastes. The surface microstructures of the wastes before and after torrefaction at a 1000 magnification are shown in Fig. 8. Hemicelluloses are branched polysaccharides containing sugar residues, such as xylose, mannose, galactose, glucose, rabinose and glucuronic acid [29]. In Fig. 8a, some branched configuration due to the biomass network of abundant hemicellulose can be clearly observed on the surface of the raw coffee residue. But the surface becomes smoother after the torrefaction, stemming from the depletion of hemicellulose from the torrefaction. For the sawdust, some microporous structure on the surface is exhibited from the torrefaction temperature of 270 °C (Fig. 8b), elucidating the structure damage of biomass by the torrefaction. In the study of Kumar et al. [30], the behavior of microporous was also found for poplar solid at various pretreatments. With regard to the rice husk, when it undergoes torrefaction at 270 °C, the porous structure can also be identified (Fig. 8c). A comparison between the sawdust and rice husk suggests that the structure damage from torrefaction on the latter is more than on the former. This may be explained by that hemicellulose contained in the rice husk is higher than in the sawdust.

4. Conclusions

Influence of torrefaction on the properties and structures on three different agricultural wastes (i.e. coffee residue, sawdust and rice husk) has been examined and compared to a high-volatile and a low-volatile coals through the proximate, elemental, fiber, calorific, thermogravimetric, SEM and FTIR analyses. By virtue of more hemicellulose contained in the coffee residue, its weight loss from torrefaction is more drastic than that of sawdust and rice husk, and this leads to a greater enhancement in its HHV. The TGA indicates that the torrefied biomass is less hygroscopic compared to its parent one and the torrefaction has a larger impact upon the lignocellulosic structure of the coffee residue. From the analysis of empirical atomic formula, it is found that the raw wastes expressed by $CH_{1.54–1.76}O_{0.65–0.89}$ will be changed to $CH_{1.02–1.57}O_{0.26–0.64}$ in the torrefied wastes This suggests that the hydrogen contained in the torrefied wastes approaches that of the high-volatile coal when the torrefaction temperature and duration increase. However, the oxygen contained in the torrefied wastes (atomic $O/C \geqq 0.26$) is still higher than that in the coals (atomic $O/C = 0.02$ and 0.08). It is also observed that torrefaction possesses the merit of reducing sulfur in biomass, implying that less sulfur oxides will be emitted if torrefied biomass is burned. From the comparison of prediction and measurement, the empirical correlation for raw biomass also enables us to predict the HHV of torrefied wastes. As a whole, within the investigated ranges of torrefaction temperature and duration, over 88% of energy yield can be obtained from the torrefaction.

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